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(54) Title: LUBRICANT COMPOSITION

(57) Abstract: The invention relates to lubricant compositions based on a base oil and a friction-reducing additive comprising a saturated aliphatic amide having a carbon chain length from 10 to 24 carbon atoms. The friction-reducing additive can also further comprise an ester or aliphatic amine. The friction-reducing additives provide enhanced oxidative stability, as compared to commercially available friction-reducing additives, in engine oils, fuels and transmission oils.

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Lubricant Composition

The present invention relates to a lubricant composition comprising a friction-reducing additive with enhanced oxidative stability.

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Lubricant compositions containing friction-reducing additives have been known for several years. Originally such compositions were used as slip gear oils, automatic transmission fluids, slideway lubricants and multipurpose tractor fluids. Such compositions made use of friction reduction to meet requirements for smooth
10 transition from static to dynamic conditions as well as reduced noise, frictional heat and start-up torque.

Then, when fuel economy became an international issue, initially, to reduce crude oil consumption, friction-reducing additives were introduced into automotive crankcase lubricants to improve fuel efficiency. In the US, additional pressure to improve fuel
15 efficiency was applied to original equipment manufacturers (OEM's) by the Corporate Average Fuel Economy (CAFE) regulation.

Following the introduction of vehicle exhaust emission regulations in various regions around the world, emphasis on friction reduction increased further. This was because it was realised that 20 - 25% of the energy generated in an engine by burning fuel
20 was lost through friction; the majority being lost at the piston liner/piston ring interface with smaller losses occurring in bearings and in the valve train. It has been predicted that, in future engines, the contribution of the piston group to engine friction will increase up to 50%.

One of the ways identified to achieve reduction in fuel consumption and emissions
25 was by suitable choice of the engine lubricant composition. Engine friction originates from several components, which operate at different conditions of load, speed and temperature. Hence, these components may experience various combinations of (elasto) hydrodynamic, mixed and boundary lubrication during engine operation. For each of these regimes, there are a number of factors that govern engine friction.

30 Investigation of those factors identified two main options to reduce friction and fuel economy. The first was to use low viscosity engine oils when fluid lubrication ((elasto) hydrodynamic regime) is the governing factor. Such fluid lubrication is especially prevalent in the bearings. The gradual reduction of engine oil viscosity over the years has already brought significant fuel savings. The second was the addition of friction-

reducing agents when boundary and/or mixed lubrication are the governing factors. Those factors are prevalent in the valve train and the piston group. In this instance, additive system design was a crucial element. Emphasis has been on selection of friction-reducing additives and control of additive/additive and additive/base fluid interactions.

Friction-reducing additives that have been used fall into three main chemically-defined categories, which are organic, metal organic and oil insoluble. The organic friction-reducing additives themselves fall within four main categories which are carboxylic acids or their derivatives, which includes partial esters, nitrogen-containing compounds such as amides, imides, amines and their derivatives, phosphoric or phosphonic acid derivatives and organic polymers. In current commercial practice examples of friction reducing additives are glycerol monooleate and oleylamide.

US 4,280,916 discloses the addition of an aliphatic monocarboxylic acid amide to a crankcase lubricant to reduce fuel consumption of internal combustion engines. The amide is preferably an unsaturated compound derived from fatty acid components of naturally occurring fats and oils.

Whilst initial fuel economy requirements, for which the above friction reducing additives were designed, focussed on the fresh oil only, new engine oil specifications have now been developed that will address fuel economy longevity as well. A good example is Sequence VI-B, an engine test, which has been developed for the ILSAC GF-3 specification. Sequence VI-B includes ageing stages of 16 and 80 hours in order to determine fuel economy as well as fuel economy longevity. These ageing stages are equivalent to 4000 – 6000 miles of mileage accumulation required prior to the EPA Metro / Highway Fuel economy test. That test is used in determining the CAFE parameter for a vehicle.

To obtain engine oil formulations that are optimised with regard to fuel economy longevity, demanding targets will be placed on base oil selection and additive system design. Those targets are to minimise the increase of viscosity thereby maintaining a low friction coefficient in the (elasto) hydrodynamic regime and to maintain low friction in the boundary and mixed lubrication regimes.

The increase of viscosity with time in the (elasto)hydrodynamic regime can be reduced or minimised by base oil selection (in terms of volatility, oxidation stability and antioxidant susceptibility) and selection of antioxidants and their treatment level.

To achieve low friction under boundary and mixed lubrication conditions, the use of effective friction-reducing additives is needed. To maintain low boundary and mixed friction over time, it is necessary to prevent consumption of these additives by processes such as oxidation and thermal breakdown. Therefore, the development of friction-reducing additives with high thermal/oxidative stability is key to meet the new requirement for high fuel economy longevity and hence a successful application in engine oil formulations.

Recent research has indicated that fuel economy can be improved further by addition of friction-reducing additives to the fuel itself. It is believed that the fuel delivers the friction-reducing additives to the piston ring-cylinder wall interface where friction is known to be high and the oil quantity is deliberately kept low. Furthermore it has been found that, as the friction-reducing additive in the fuel accumulates in the engine oil, friction is also reduced in oil-lubricated parts.

The current range of engine friction-reducing additives was not designed to meet the above-mentioned combination of fuel economy and fuel economy longevity requirements for friction-reducing additives. It is known that both glycerol monooleate and oleylamide are susceptible to oxidative breakdown over time. Furthermore, there is another disadvantage in the use of oleylamide as it has low compatibility with the base oils currently being used.

Surprisingly, it has been found that specific saturated aliphatic amides and blends of such amides with esters meet the new demands for friction-reducing additives with respect to oxidative stability without compromise to their friction-reducing properties. According to the present invention, a lubricant composition for use in fuel, engine oils and transmission oil comprises a major component selected from a fuel or a base oil and a friction-reducing additive which comprises a saturated aliphatic amide having a carbon chain length ranging from 10 to 24 carbon atoms.

The carbon chain of the saturated aliphatic amide may be branched or straight chained or comprise an aliphatic ring. Preferably the saturated aliphatic amide is derived from a fatty acid. Preferably the saturated aliphatic amide is a primary amide. The primary amide may be derived from ammonolysis of an ester, acid chloride or

acid anhydride. Examples of the saturated aliphatic amide include capryl amide, lauryl amide, myristyl amide, palmityl amide, stearyl amide, isostearyl amide, arachidyl amide, behenyl amide and lignoceryl amide. Preferably the carbon chain length ranges from 12 to 22 carbon atoms, more preferably from 14 to 20 carbon atoms Preferred examples of the saturated aliphatic amide are myristyl amide, 5 stearyl amide and isostearyl amide with isostearyl amide being the most preferred.

The friction-reducing additive may further comprise an ester.

10 The ester may be derived by direct esterification of an acid, acid chloride or acid anhydride with an alcohol or it may be derived by transesterification of an ester with an alcohol. Preferably it is derived by direct esterification of at least one acid with at least one alcohol.

15 The at least one alcohol, from which the ester is derived, may be a monohydric or a polyhydric alcohol. Preferably it is a polyhydric alcohol. Examples of suitable polyhydric alcohols include ethylene glycol, propylene glycol, trimethylene glycol, diols of butane, neopentyl glycol, trimethylol propane and its dimer, pentaerythritol and its dimer, glycerol, inositol and sorbitol. Trimethylol propane and glycerol are 20 specifically preferred.

The at least one alcohol may be partially or fully esterified. Preferably it is partially esterified.

25 The at least one monocarboxylic acid is preferably a fully saturated acid, but it may contain up to 25% of unsaturation, for example coconut oil which has up to 12% unsaturated acids. The at least one monocarboxylic acid has a carbon chain which may be straight chained or branched or comprise an aliphatic ring. Preferably the carbon chain length ranges from 12 to 22 carbon atoms, more preferably 14 to 20 30 carbon atoms and specifically 18 carbon atoms. Examples of suitable monocarboxylic acids include coconut fatty acid, lauric acid, myristic acid, palmitic acid, stearic acid, isostearic acid, hydrogenated C18 monomeric acid, C18 monomeric acid, arachidic acid, behenic acid and lignoceric acid and mixtures thereof. Hydrogenated C18 monomeric acid is the C18 monomer by-product resulting 35 from the dimerisation of oleic acid, which is then subsequently hydrogenated. C18 monomeric acid is the C18 monomer by-product itself, which typically is about 20% unsaturated.

The ratio of saturated aliphatic amide in the blend to ester ranges from 95: 5 wt % to 5 to 95 wt %, more preferably from 80:20 to 20:80.

5 The friction-reducing additive according to the invention is present at levels between 0.01 and 5% by weight, more preferably between 0.1 and 3%, even more preferably between 0.25 and 2% in the lubricant composition.

The base oil of the lubricant composition may be chosen from mineral oils, poly α olefins, alkylbenzenes, monoesters, diesters, polyol esters, complex esters, polyalkylene glycols and mixtures thereof. Preferably the base oil comprises a
10 mineral oil or a poly α olefin.

The kinematic viscosity at 100 °C for the base oil should be chosen such that it allows the formulation of low viscosity, fuel-efficient oils. This ranges from 1 to 20, preferably 3 to 8, and particularly 4 to 6, cSt.

15 The lubricant composition may also comprise other additives of known functionality at levels between 10 to 20, %, more preferably between 12 to 18, % more especially between 14 to 16, % of the total weight of the lubricant composition. Suitable additives include detergents, dispersant, antiwear/extreme pressure additives,
20 viscosity modifiers, anticorrosion additives, antifoam, pour point depressants and the like.

According to a further embodiment of the present invention use of a lubricant composition which comprises a major component selected from a fuel or a base oil and a friction- reducing additive which comprises a saturated aliphatic amide having
25 a carbon chain length ranging from 10 to 24 carbon atoms in an engine oil, fuel or transmission oil.

For use of the lubricant composition the friction-reducing additive may further comprise an ester.

30 The lubricant composition is designed for use in fuel and both engine and transmission oils. By fuel it is meant both gasoline and diesel fuel.

By engine oils it is meant both gasoline and diesel (including heavy duty diesel) engine oils. By transmission oils it is meant automatic, gear, rear axle and
35 continuously variable transmission oils.

The invention will now be described further by way of example only with reference to the following Examples.

Example 1

- 5 The oxidative stability of friction-reducing additives in gasoline engine oils was tested as follows:

The induction time in minutes, i.e. the time up to when oxidation of the friction reducing additive starts, was measured by high pressure differential scanning calorimetry (DSC) of various friction-reducing additives, each with 0.5% by weight
10 antioxidant (Irganox L115 - ex Ciba Speciality Chemicals) present, using a Mettler DSC27HP with a Mettler TC 15 TA controller, under the following test conditions:

Start temperature: 30°C.

Heating rate: 50°C/min till test temperature of 170°C.

- 15 Air pressure: 40 bar Airflow: 50ml/min

Sample quantity: 5 +/- 0.4mg.

Crucible: Aluminium type, 40µl.

The results are illustrated in Table 1 below.

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Table 1

Friction-Reducing Additive	Induction Time (Minutes)
Isostearylamine	43
Stearylamine	71
Isostearylamine/ Glycerolmonoistearate blend (50:50 percentage wt/wt)	42
Stearylamine/ Glycerolmonoistearate (20:80 percentage wt/wt)	50
Oleylamine (Comparative)	11
Glycerolmonooleate (Comparative)	4

- The data in Table 1 clearly illustrates that friction-reducing additives according to the present invention have significantly enhanced oxidative stability as compared to the
25 commercially available friction-reducing additives specified.

Example 2

The coefficient of friction of a lubricant composition comprising 0.5% by weight of friction-reducing additive was determined over a temperature range of 40 to 140°C using a pin-on-ring tribometer. The ring is a 100Cr6 stainless steel ring of 730mm diameter and the pin is a cylinder of the same material of 8mm diameter, the pin having flexible ends so that each end can bend slightly to allow full alignment with the ring. The load applied was 100N and the speed of rotation was 0.03m/s to ensure that the system operates under boundary lubrication. The results are illustrated in Tables 2, 3, 4 and 5 below where CEC RL 179/2 is a 5W-30 calibration formulated engine oil for the European fuel economy test, number CEC L-54-T-96, ASTM 1007 is a 5W-30 formulated engine reference oil for the API (American Petroleum Institute) Sequence VIB fuel economy test, OR-F53244Q-A-01 oil is a 5W-40 formulated engine oil formulation from Chevron Oronite and an 0W/30 formulated oil based on hydrogenated oligomers of decene-1

Table 2

Formulated Base Oil	CEC RL 179/2	CEC RL 179/2	CEC RL 179/2	CEC RL 179/2
Friction Reducing Additive	Not present	Glycerol Monooleate (Comparative)	Oleylamide (Comparative)	Isostearylamide
Temperature [°C]	friction coefficient	Friction coefficient	friction coefficient	Friction coefficient
40	0.1127	0.0959	0.0885	0.0980
50	0.1179	0.0958	0.0882	0.0981
60	0.1206	0.0913	0.0878	0.0966
70	0.1220	0.0875	0.0875	0.0958
80	0.1232	0.0904	0.0888	0.0956
90	0.1243	0.1008	0.0946	0.0977
100	0.1255	0.1084	0.1025	0.1012
110	0.1268	0.1137	0.1074	0.1071
120	0.1286	0.1177	0.1101	0.1075
130	0.1336	0.1200	0.1109	0.1072
140	0.1349	0.1192	0.1077	0.1048

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Table 3

Formulated Base Oil	ASTM 1007	ASTM 1007	ASTM 1007	ASTM 1007
Friction Reducing Additive	Not Present	Glycerol Monooleate (Comparative)	Oleylamide (Comparative)	Isostearylamide
Temperature	friction coefficient	Friction coefficient	friction coefficient	friction coefficient
[°C]				
40	0.1219	0.1132	0.0952	0.0976
50	0.1285	0.1181	0.0992	0.1019
60	0.1356	0.1235	0.1019	0.1056
70	0.1400	0.1277	0.1044	0.1073
80	0.1420	0.1303	0.1074	0.1099
90	0.1425	0.1317	0.1101	0.1121
100	0.1422	0.1341	0.1132	0.1146
110	0.1415	0.1338	0.1175	0.1165
120	0.1401	0.1353	0.1207	0.1188
130	0.1386	0.1352	0.1239	0.1211
140	0.1352	0.1336	0.1251	0.1222

5 Table 4

Formulated Base Oil	OR-F53244Q-A-01	OR-F53244Q-A-01	OR-F53244Q-A-01	OR-F53244Q-A-01
Friction Reducing Additive:	Not present	Glycerol Monooleate (Comparative)	Oleylamide (Comparative)	Isostearylamide
Temperature	friction coefficient	Friction coefficient	friction coefficient	friction coefficient
[°C]				
40	0.1234	0.1028	0.1088	0.1143
50	0.132	0.1056	0.1107	0.1172
60	0.1304	0.1058	0.1118	0.1175
70	0.1344	0.1101	0.1130	0.1170
80	0.1394	0.1199	0.1151	0.1176
90	0.1408	0.1258	0.1175	0.1201
100	0.1463	0.1308	0.1198	0.1220
110	0.1504	0.1369	0.1215	0.1239
120	0.1545	0.1419	0.1230	0.1263
130	0.1588	0.1468	0.1260	0.1285
140	0.1638	0.1514	0.1279	0.1333

Table 5

Formulated Base Oil	0W/30	0W/30	0W/30	0W/30	0W/30
Friction Reducing Additive:	Not present	Glycerol Monooleate (Comparative)	Oleylamide (Comparative)	Isostearyl amide/ Glycerol monoisostearate (50:50)	Stearyl amide/ Glycerol Mono Isostearate (20:80)
Temp. [°C]	friction coefficient	friction coefficient	Friction coefficient	friction coefficient	friction coefficient
40	0.1368	0.1066	0.1110	0.1084	0.1120
50	0.1395	0.1063	0.1110	0.1103	0.1121
60	0.1410	0.1098	0.1146	0.1117	0.1119
70	0.1426	0.1140	0.1134	0.1123	0.1118
80	0.1436	0.1154	0.1105	0.1123	0.1115
90	0.1442	0.1164	0.1093	0.1123	0.1111
100	0.1445	0.1164	0.1078	0.1125	0.1108
110	0.1449	0.1173	0.1071	0.1122	0.1107
120	0.1456	0.1195	0.1078	0.1125	0.1112
130	0.1466	0.1227	0.1090	0.1134	0.1133
140	0.1545	0.1302	0.1119	0.1240	0.1200

- It is clear from the data in Tables 2, 3, 4 and 5 that friction-reducing additives of the present invention have similar friction-reducing capabilities to the known friction-reducing additives of glycerol monooleate and oleylamide. Although the friction-reducing additives of the present invention have enhanced oxidative stability as compared to known friction-reducing additives (as illustrated in Example 1 above) there is no corresponding detrimental effect on their friction-reducing properties.

Example 3

- Compatibility of isostearyl amide as the friction-reducing additive at 0.5% by weight with the base oil was determined as follows. The friction-reducing additive was added to the base oil and the mixture heated to about 80°C whilst stirring and kept at that temperature for 30 minutes to ensure complete solubilisation. After cooling to ambient temperature (about 23°C) the mixture was stored for a maximum of 3 months and periodically checked for compatibility. The results are shown in Table 6 where CEC RL 179/2 is the base oil and Table 7 where ASTM 1007 is the base oil. By compatible it is meant that the fluid is bright and clear and does not show any sign of haziness, turbidity and precipitation.

By non-compatible it is meant that the fluid is not bright and clear and shows signs of haziness and/or turbidity and/or precipitation.

Table 6

Friction-Reducing Additive (wt %)	After One Day	After 5 Days	After 10 Days	After 15 Days
0.5% Isostearylamine	Compatible	Compatible	Compatible	Compatible
0.5% Oleylamine (Comparative)	Not Compatible	Not Compatible	Not Compatible	Not Compatible

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Table 7

Friction-Reducing Additive (wt %)	After One Day	After 5 Days	After 10 Days	After 15 Days
0.5% Isostearylamine	Compatible	Compatible	Compatible	Compatible
0.5% Oleylamine (Comparative)	Not Compatible	Not Compatible	Not Compatible	Not Compatible

It is clear from the data in Tables 6 and 7 that isostearylamine of the present invention is compatible with the base oils at ambient temperature.

10 Example 4

The compatibility of isostearylamine and a blend of isostearylamine with glycerol monoisostearate (50:50 weight percentage) as the friction-reducing additive at 0.5% by weight with the 0W/30 formulated base oil were determined as follows. The friction-reducing additive was added to the formulated base oil and the mixture
15 cooled to -10°C. The mixture was stored for a maximum of 3 months and periodically checked for compatibility. By compatible it is meant that the fluid is bright and clear and does not show any sign of haziness, turbidity and precipitation.

The isostearylamine/glycerol monoisostearate blend was found to be compatible with
20 the formulated base oil for the whole test period. For the isostearylamine itself crystallization and sedimentation started to occur on day 7 whereas for a comparative sample containing oleylamine, crystallisation and sedimentation started to occur on day 2 of the test period.

It is clear from the above data that friction modifiers in accordance with the present invention have enhanced compatibility with the formulated base oils as compared to oleylamide.

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Claims

1. A lubricant composition for use in fuel, engine oil and transmission oil comprises as a major component selected from a fuel or a base oil and a friction- reducing additive which comprises a saturated aliphatic amide having a carbon chain length ranging from 10 to 24 carbon atoms.
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2. A lubricant composition as claimed in claim 1 wherein the amide is selected from capryl amide, lauryl amide, myristyl amide, palmityl amide, stearyl amide, isostearyl amide, arachidyl amide, behenyl amide and lignoceryl amide.
10
3. A lubricant composition as claimed in claim 1 wherein the friction-reducing additive further comprises an ester.
4. A lubricant composition as claimed in claim 3 wherein the ester has a carbon chain length ranging from 10 to 24 carbon atoms.
15
5. A lubricant composition as claimed in either claim 3 or claim 4 wherein the ratio of saturated aliphatic amide to ester ranges from 95: 5 wt % to 5 to 95 wt %.
20
6. A lubricant composition as claimed in any of claims 1 to 5 wherein the friction-reducing additive is present at levels between 0.01 and 5% by weight.
7. A lubricant composition for use in fuel, engine oil and transmission oil, which comprises as a major component thereof a base oil and isostearyl amide.
- 25 8. Use of a lubricant composition which comprises a major component selected from a fuel and a base oil and a friction- reducing additive which comprises a saturated aliphatic amide having a carbon chain length ranging from 10 to 24 carbon atoms in an engine oil.
- 30 9. Use of a lubricant composition which comprises a major component selected from a fuel and a base oil and a 'friction- reducing additive which comprises a saturated aliphatic amide having a carbon chain length ranging from 10 to 24

carbon atoms in a fuel.

5 10. Use of a lubricant composition which comprises a major component selected from a fuel and a base oil and a friction-reducing additive which comprises a saturated aliphatic amide having a carbon chain length ranging from 10 to 24 carbon atoms in a transmission oil.

10 11. Use of a lubricant composition as claimed in any one of claims 8 to 10 wherein the friction-reducing additive further comprises an ester.

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(54) Title: LUBRICANT OR FUEL COMPOSITION COMPRISING AN AMIDE AS FRICTION-REDUCING ADDITIVE

(57) Abstract: The invention relates to lubricant compositions based on a base oil and a friction-reducing additive comprising a saturated aliphatic amide having a carbon chain length from 10 to 24 carbon atoms. The friction-reducing additive can also further comprise an ester or aliphatic amine. The friction-reducing additives provide enhanced oxidative stability, as compared to commercially available friction-reducing additives, in engine oils, fuels and transmission oils.

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IPC 7 C10L C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	US 4 280 916 A (RICHARDS HARRY F ET AL) 28 July 1981 (1981-07-28) cited in the application abstract column 1, line 38 -column 2, line 36 column 6, line 6 -column 6, line 15 example I table I ---	1-3,5,6, 8,11
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☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

10 April 2003

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INTERNATIONAL SEARCH REPORT

Intern: Application No

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